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Paints and varnishes — Determination of the percentage volume of nonvolatile matter

Part 2: Method using the determination of non-volatile-matter content in accordance with ISO 3251 and determination of dry film density on coated test panels by the Archimedes principle



National foreword

This British Standard is the UK implementation of EN ISO 3233-2:2014.

The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Paints and varnishes - Determination of the percentage volume of non-volatile matter - Part 2: Method using the determination of non-volatile-matter content in accordance with ISO 3251 and determination of dry film density on coated test panels by the Archimedes principle (ISO 3233-2:2014)

Peintures et vernis - Détermination du pourcentage en volume de matière non volatile - Partie 2: Méthode utilisant la teneur en matière non volatile déterminée conformément à l'ISO 3251 et la masse volumique du feuil sec déterminée par le principe d'Archimède sur des panneaux d'essai revêtus (ISO 3233-2:2014)

Beschichtungsstoffe - Bestimmung des Volumens nichtflüchtiger Anteile - Teil 2: Verfahren mit Bestimmung des Gehaltes an nichtflüchtigen Anteilen nach ISO 3251 und Bestimmung der Trockenfilmdichte beschichteter Probenplatten nach dem Archimedes-Prinzip (ISO 3233-2:2014)

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Foreword

This document (EN ISO 3233-2:2014) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes" the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2015, and conflicting national standards shall be withdrawn at the latest by January 2015.

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The text of ISO 3233-2:2014 has been approved by CEN as EN ISO 3233-2:2014 without any modification.

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Foreword

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The committee responsible for this document is ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

ISO 3233 consists of the following parts, under the general title *Paints and varnishes* — *Determination of the percentage volume of non-volatile matter*:

- Part 1: Method using a coated test panel to determine non-volatile matter and to determine dry film density by the Archimedes principle
- Part 2: Method using the determination of non-volatile-matter content in accordance with ISO 3251 and determination of dry film density on coated test panels by the Archimedes principle
- Part 3: Determination by calculation from the non-volatile-matter content determined in accordance with ISO 3251, the density of the coating material and the density of the solvent in the coating material

Paints and varnishes — Determination of the percentage volume of non-volatile matter —

Part 2:

Method using the determination of non-volatile-matter content in accordance with ISO 3251 and determination of dry film density on coated test panels by the Archimedes principle

1 Scope

This part of ISO 3233 specifies a method for determining the non-volatile matter by volume (NV $_{v}$) of coating materials by determining the practical dry-film density. This method determines the volume percentage of non-volatile matter in paints, varnishes and related products by measuring the density of a dry coating for any specified temperature range and period of drying or curing.

Using the non-volatile matter by volume results obtained in accordance with this part of ISO 3233, it is possible to calculate the practical spreading rate of coating materials.

This method specifies an additional shape of plate to those described in ISO 3233-1 and is suitable for all products which can be applied by dipping.

This part of ISO 3233 is not applicable to coating materials which exceed the critical pigment volume concentration (CPVC).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1513, Paints and varnishes — Examination and preparation of test samples

ISO 2808, Paints and varnishes — Determination of film thickness

ISO 2811 (all parts), Paints and varnishes — Determination of density

ISO 3251, Paints, varnishes and plastics — Determination of non-volatile-matter content

ISO 15528, Paints, varnishes and raw materials for paints and varnishes — Sampling

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

non-volatile matter

NV

residue by mass obtained by evaporation under specified conditions

[SOURCE: ISO 4618:2006, 2.161]

3.2

non-volatile matter by volume

NV_{V}

percentage residue by volume obtained by evaporation under specified conditions

33

spreading rate

S

surface area that can be covered by a given quantity of coating material to give a dried film of requisite

Note 1 to entry: It is expressed in m²/l or m²/kg.

3.4

practical spreading rate

 $S_{\mathbf{n}}$

spreading rate which is obtained in practice on the particular substrate being coated

3.5

practical dry-film density

 $\rho_{\rm p}$

practically determined density of a dried and cured coating

4 Principle

The non-volatile matter by volume is calculated from the quotient of the density of the coating material and the dry film, with the dry-film density being determined practically.

5 Apparatus

Standard laboratory apparatus together with the following:

5.1 Metal plate, (40 ± 1) mm × (85 ± 1) mm, with a small hole 2 mm to 3 mm from the edge. A plate with a tip on one of the shorter edges is easier to immerse in the coating material (see Figure 1).

The material of the plate shall be suitable and adapted to the coating material under test. In addition the material of the plate shall not change its volume during contact with the coating material under test. The thickness of the plate is about 0,7 mm or it shall be agreed between the interested parties.

Smaller plates may be used subject to agreement between the interested parties, provided that the coated surface area is at least $5\,600\,\text{mm}^2$.

Dimensions in millimetres

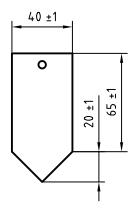


Figure 1 — Suitable plate for immersion

- **5.2 Hook**, made of stainless material or synthetic thread, for attaching the plate to the balance during weighing operations. The diameter of the wire shall not exceed 0,30 mm because of surface tension effects.
- **5.3 Beaker**, of a size convenient for immersing the plate with a clearance of at least 10 mm and which can be accommodated in the balance case.
- **5.4 Analytical balance**, accurate to 0,1 mg. A single-pan balance is most convenient, and a useful modification is to replace the balance pan by a standard counterweight attachment.
- **5.5 Support**, for holding the beaker under the balance stirrup without jamming the pan damper, if a counterweight as recommended in <u>5.4</u> is not available.
- **5.6 Immersion liquid** of suitable density, in which the plate is immersed.

NOTE Water is a suitable immersion liquid for most coating materials. Other organic liquids are also suitable provided they do not attack the coating.

- **5.7 Desiccator** containing a suitable desiccant.
- **5.8 Air oven**, capable of maintaining the specified or agreed test temperature to $\pm 2.0^{\circ}$ C (for temperatures up to 150°C) or $\pm 3.5^{\circ}$ C (for temperatures above 150°C and up to 200°C). An air oven with forced ventilation shall be used.

WARNING — To protect against explosions and fire, careful handling of products containing flammable volatile materials is essential.

Drying in a vacuum can be beneficial for certain applications. In such cases the conditions shall be agreed. Air ovens of the same type shall be used by all parties for referee tests.

6 Sampling

Take a representative sample of the coating material to be tested, in accordance with ISO 15528.

Examine and prepare the samples for testing in accordance with ISO 1513.

7 Procedure

7.1 Number of determinations and preparation

Carry out the determination in duplicate.

Degrease and clean the plate (5.1). Dry the plate and hook at the specified temperature for 10 min, and cool in the desiccator.

7.2 Determination of the practical dry-film density

7.2.1 Determination of the mass of the uncoated plate in air and in the immersion liquid

Weigh the cleaned and dried plate plus hook in air to an accuracy of 1 mg (m_1) .

Then place the plate in the beaker with the immersion liquid, e.g. water, and weigh to an accuracy of 1 mg (m_2). Ensure that the plate is always immersed to the same depth, with the liquid about 10 mm above the top of the plate. There shall be no air bubbles at any place on the plate (see Figure 2).

NOTE If water is used as the immersion liquid, it is beneficial to add 1 or 2 drops of a wetting agent to ensure thorough wetting of the plate.

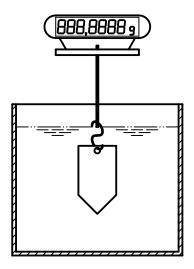


Figure 2 — Weighing the plate

7.2.2 Weighing the coating material and determination of the mass of the plate with coating material

The coating material shall always be ready to use when it is tested.

The dry-film thickness on the plate shall correspond approximately to the dry-film thickness of the coating material used in practice.

In the case of thixotropic or high viscous coating materials they may be diluted in accordance with the manufacturer's instructions until the sample is uniformly spread over the plate.

The preferred method of coating the plate is to immerse it in the coating material. Withdraw it at a steady rate and remove any excess coating material by, for example, drawing a glass rod along the lower edge of the plate. No thick edge shall be allowed to form on the lower edge. If any air bubbles form on the coated surfaces, burst them with a needle.

Dry/cure the coated plate product-specifically in accordance with the instructions of the manufacturer of the coating material under test.

Cool the coated plate to room temperature after drying/curing in the desiccator.

After cooling, weigh the coated plate in air to an accuracy of 1 mg (m_3) .

Then place the coated plate in the beaker with the immersion liquid, e.g. water, and weigh to an accuracy of 1 mg (m_4). Ensure that the coated plate is always immersed to the same depth, with the liquid about 10 mm above the top of the plate. There shall be no air bubbles at any place on the coated plate.

7.3 Determination of the density

The density of the immersion liquid (ρ_1) and the coating material (ρ_2) is determined to an accuracy of 0,001 g/cm³ in accordance with one of the methods specified in ISO 2811 (all parts).

7.4 Determination of the non-volatile-matter content

Determine the non-volatile-matter content in accordance with ISO 3251.

7.5 Determination of film thickness

Determine the dry film thickness using one of the methods described in ISO 2808.

8 Evaluation

8.1 Calculation of the practical dry-film density

Calculate the practical dry-film density (ρ_p), in grams per cubic centimetre, as follows using the determined mass values and the density of the immersion liquid:

$$\rho_{\rm p} = \frac{m_3 - m_1}{m_2 + m_3 - m_1 - m_4} \cdot \rho_1 \tag{1}$$

where

 m_1 is the mass of the uncoated plate weighed in air, in grams;

 m_2 is the mass of the uncoated plate weighed in the immersion liquid, in grams;

 m_3 is the mass of the coated plate weighed in air, in grams;

 m_4 is the mass of the coated plate weighed in the immersion liquid, in grams;

 ρ_1 is the density of the immersion liquid, in grams per cubic centimetre.

8.2 Calculation of the non-volatile matter by volume using the practical dry-film density

The conversion below yields the following for the practical determination of the percentage non-volatile matter by volume, $NV_{V,\,p}$:

$$NV_{V, p} = NV \cdot \frac{\rho_2}{\rho_1} \left(\frac{m_2 + m_3 - m_1 - m_4}{m_3 - m_1} \right) = NV \cdot \frac{\rho_2}{\rho_p}$$
 (2)

where

NV is the non-volatile matter of the coating material, as a percentage by mass;

 m_1 is the mass of the uncoated plate weighed in air, in grams;

*m*₂ is the mass of the uncoated plate weighed in the immersion liquid, in grams;

 m_3 is the mass of the coated plate weighed in air, in grams;

 m_4 is the mass of the coated plate weighed in the immersion liquid, in grams;

 $\rho_{\rm p}$ is the practical dry-film density, in grams per cubic centimetre;

 ρ_1 is the density of the immersion liquid, in grams per cubic centimetre;

 ρ_2 is the density of the coating material, in grams per cubic centimetre.

8.3 Determination of the practical spreading rate

The practical spreading rate (s_p) is a value which is calculated solely from the non-volatile matter by mass or by volume.

The practical spreading rate is the quotient of the surface area coated and the mass required for this, in square metres per kilogram or the volume, in square metres per litre.

Calculate the practical spreading rate relative to the mass ($s_{p, m}$), using Formula 3:

$$s_{\mathrm{p, m}} = \frac{A}{m_0} = \frac{\mathrm{NV}}{t_{\mathrm{d}} \cdot \rho_{\mathrm{p}}} \cdot 10 \tag{3}$$

Calculate the practical spreading rate relative to the volume $(s_{p, V})$, using Formula 4:

$$s_{p, V} = \frac{A}{V_0} = \frac{NV \cdot \rho_2}{t_d \cdot \rho_p} \cdot 10 = \frac{NV_{V,p}}{t_d} \cdot 10$$
(4)

where

A is the coated surface area, in square metres;

 m_0 is the mass required for coating, in kilograms;

 V_0 is the volume required for coating, in litres;

NV is the non-volatile matter of the coating material, as a percentage by mass;

NV_{V,p} is the practical non-volatile matter by volume, as a percentage by volume;

t_d is the dry-film thickness of the coating, in micrometres;

 ho_{p} is the practical dry-film density, in grams per cubic centimetre;

 ρ_2 is the density of the coating material, in grams per cubic centimetre.

9 Precision

9.1 Repeatability

The repeatability limit r is the value below which the absolute difference between two test results (each being the average of two valid determinations) of this test method can be expected under similar conditions. The test results shall be determined on the same test material by the same test technician in the same laboratory within a short period of time in accordance with the standard test method.

Two results of the non-volatile matter by volume calculated on the basis of the theoretical dry-film density are regarded as acceptable and in compliance with the standard for the repeatability limit if they do not differ by more than the following value: $0.48 + (0.0086 \times NV_v)$.

9.2 Reproducibility

The reproducibility limit *R* is the value below which the absolute difference between two test results (each being the average of two valid determinations) of this test method can be expected under matching conditions. The test results shall be determined on the same test material by different test technicians in different laboratories in accordance with the standard test method.

Two results of the non-volatile matter by volume calculated on the basis of the theoretical dry-film density are regarded as acceptable and in compliance with the standard for the reproducibility limit if they do not differ by more than the following value: $1,06 + (0,009 \text{ 6} \times \text{NV}_v)$.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested (manufacturer, product code, batch number etc.);

- b) a reference to this part of ISO 3233 (ISO 3233-2);
- c) an indication of the test method used for the dry-film density;
- d) the type of plate used;
- e) the immersion liquid;
- f) the type of air oven used;
- g) the application method for the sample coating on the plate, including drying/curing conditions for the coating;
- h) the dry-film thickness of the coating, in micrometres;
- i) the result of the test, as specified in <u>Clause 8</u>;
- j) any deviation from the specified test method;
- k) any unusual features (anomalies) observed during the test;
- l) the date of testing.

Annex A

(informative)

Overview of existing methods for determination of non-volatile matter content and volume of non-volatile matter

Standard	Result	Determined	Calculated
		(practical)	(theoretical)
ISO 3233-1	Practical percentage volume of non-volatile matter NV _V	Mass of the uncoated disc or plate in air m_1	Practical dry-film density $\rho_{\rm p}$ of the test portion, mean value of 3 determinations
		Mass of the uncoated disc or plate immersed in the immersion liquid m_2	Non-volatile-matter content NV $_{m}$ a of the test portion, mean value of 3 determinations
		Mass of the wet coated disc or plate m_3	Non-volatile matter by volume NV_V , calculated from the mean values above
		Mass of the dry coated disc or plate in air m_4	Spreading rate relative to the mass $s_{\rm m}$ Spreading rate relative to the volume $s_{\rm V}$
		Mass of the dry coated disc or plate immersed in the immersion liquid m_5	
		Density of immersion liquid $ ho_1$	
		Density of the coating material $ ho_2$	
ISO 3233-2	Practical percentage volume of non-volatile matter NV _{V,p}	Mass of the uncoated plate in air m_1	Practical dry-film density $\rho_{\rm p}$ of the test portion, 2 determinations
		Mass of the uncoated plate immersed in the immersion liquid	Practical non-volatile matter by volume NV _{V,p} , mean value of 2 determinations
		m_2 Mass of the dry coated plate in air m_3 Mass of the dry coated plate immersed in the immersion liquid m_4	Practical spreading rate relative to the mass $s_{\rm p,m}$ Practical spreading rate relative to the volume $s_{\rm p,V}$
		Density of immersion liquid $ ho_1$	
		Density of the coating material $ ho_2$	
		NV of the coating material in accordance with ISO 3251	
ISO 3233-3	Theoretical percentage volume of non-volatile matter NV _{V,t}	NV of the coating material in accordance with ISO 3251	Theoretical dry-film density $ ho_{t}$ of the test portion, single determination
		Density of the coating material $ ho_1$	Theoretical non-volatile matter by volume $NV_{V,t}$
		Density of the solvents in the coating material $ ho_2$	Theoretical spreading rate relative to the mass $s_{t,m}$
			Theoretical spreading rate relative to the volume $s_{t,V}$

Table A.1 (continued)

Standard	Result	Determined	Calculated
		(practical)	(theoretical)
ISO 3251	Non-volatile- matter content NV	Masses of the empty dish m_1 , dish with the wet coating material m_2 and dish with the residue after drying m_3 Mean value of duplicates	Percentage by mass of non-volatile-matter content NV

The subscript "m" has been introduced because ISO 3233-1 and ISO 3251 specify different conditions for the determination of NV.

Bibliography

- [1] ISO 4618:2006, Paints and varnishes Terms and definitions
- [2] ISO 3233-1, Paints and varnishes Determination of the percentage volume of non-volatile matter Part 1: Method using a coated test panel to determine non-volatile matter and to determine dry film density by the Archimedes principle
- [3] ISO 3233-3, Paints and varnishes Determination of the percentage volume of non-volatile matter Part 3: Determination by calculation from the non-volatile-matter content determined in accordance with ISO 3251, the density of the coating material and the density of the solvent in the coating material





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